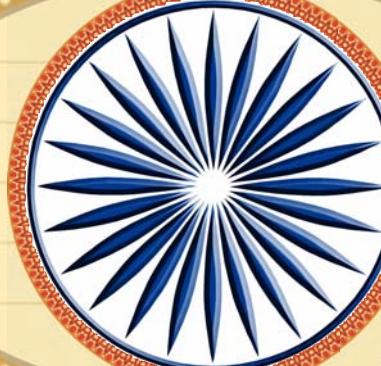




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मानक



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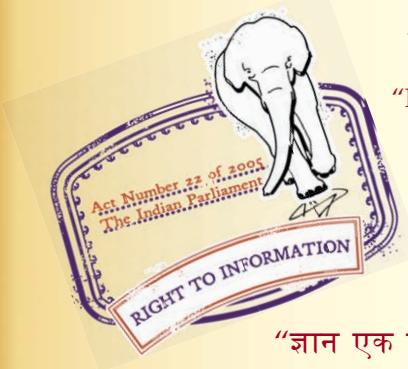
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IS 3986 (1988): Sodium Lauryl Sulphate for Cosmetic Industry [PCD 19: Cosmetics]



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Indian Standard

**SPECIFICATION FOR
SODIUM LAURYL SULPHATE
FOR COSMETIC INDUSTRY**

(Third Revision)

First Reprint DECEMBER 1991

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**BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002**

AMENDMENT NO. 1 APRIL 2002
TO
IS 3986 : 1988 SPECIFICATION FOR SODIUM LAURYL
SULPHATE FOR COSMETIC INDUSTRY

(Third Revision)

[*Page 2, Table 1, Sl No. (viii)*] — Substitute the following for the existing:

(2)	(3)	(4)	(5)
'pH (1 percent solution)	7.5-10.5	7.5-10.5	7.5-11.0'

(*Page 6, clause A-7*) — Substitute the following for the existing:

A-7 DETERMINATION OF SODIUM SULPHATE

A-7.1 Reagents

A-7.1.1 Dithiozone Solution — 0.05% m/v in acetone.

A-7.1.2 Nitric acid — 1M.

A-7.1.3 Acetone

A-7.1.4 Lead Nitrate — 0.01M : 3.312 g diluted to 1000 ml with water.

A-7.1.5 Dichloroacetic Acid Solution — Dilute 67 ml of dichloroacetic acid to 300 ml with water and neutralize to litmus paper using 10M ammonia. Cool, add 33 ml of Dichloroacetic acid and dilute with water to 600 ml.

A-7.2 Procedure

Dissolve 0.5 g of the material in 20 ml of water, warming gently if necessary and add 1 ml of 0.05% m/v solution of dithiozone in acetone. If the solution is red, add 1M Nitric acid, dropwise, until it becomes bluish green. Add 2 ml of dichloroacetic acid solution and 80 ml of acetone and titrate with 0.01M lead nitrate until permanent orange-red colour is obtained.

Amend No. 1 to IS 3986 : 1988

A-7.3 Calculation

$$\text{Sodium Sulphate(%) = } \frac{V \times N \times 0.00142 \times 100}{0.01 \times M}$$

where

V = ml of lead nitrate required,

N = molarity of lead nitrate solution, and

M = mass of sample taken for test.

(PCD 19)

**AMENDMENT NO. 2 JULY 2004
TO
IS 3986 : 1988 SPECIFICATION FOR
SODIUM LAURYL SULPHATE FOR COSMETIC
INDUSTRY**

(Third Revision)

[*Page 2, Table 1, Sl No. (ii)*] — Substitute the following for the existing:

Sl No.	Characteristic	Requirement			Methods of Test (Ref to Cl No. in Appendix A)
		Type 1	Type 2	Type 3	
(1)	(2)	(3)	(4)	(5)	(6)
ii)	Unsulphated fatty alcohols, percent by mass, <i>Max</i>	1.0	2.5	1.1	A-3

(PCD 19)

Reprography Unit, BIS, New Delhi, India

Indian Standard

**SPECIFICATION FOR
SODIUM LAURYL SULPHATE
FOR COSMETIC INDUSTRY**

(Third Revision)

0. FOREWORD

0.1 This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards on 10 August 1988, after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1966 and subsequently revised in 1973, prescribing three types of sodium lauryl sulphate. In the second revision (1981), requirement for the sum of actual percent contents of sodium chloride, sodium sulphate, unsulphated fatty alcohols and moisture was deleted and requirement of pH included.

0.2.1 In this revision, on the basis of experience gained in the industry, requirement for sodium lauryl sulphate for Type 1 is being revised from 97 percent to 94 percent, minimum. Sodium

sulphate for Type 2 is being revised from 4 to 6 percent, maximum. Requirement of moisture content has been deleted and description clause has been suitably amplified.

0.3 Sodium lauryl sulphate [$\text{CH}_3(\text{CH}_2)_10\text{CH}_2\text{O}.\text{SO}_3\text{Na}$], mainly used as a surfactant and foaming agent, finds use in dental creams, shampoos and cosmetic emulsions, in general.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements, and methods of sampling and test for sodium lauryl sulphate for cosmetic industry.

ing to methods given in Appendix A. Reference to relevant clauses of Appendix A is given in col 6 of Table 1.

2. TYPES

2.1 There shall be three types of the material differing in the content of sodium lauryl sulphate (see Table 1). Type 1 and Type 2 are solid type materials and Type 3 is liquid material.

4. PACKING AND MARKING

4.1 **Packing** — Unless otherwise agreed to between the purchaser and the supplier, the solid type material (Types 1 and 2) shall be packed in mild steel drums lacquered from inside or HDPE woven bags while the liquid material (Type 3) shall be packed in HDPE carboys.

4.2 **Marking** — The containers shall be securely closed and marked with the name, type and mass of the material in the container; molecular mass of the fatty alcohol; manufacturer's name and trade-mark, if any; batch number in code or otherwise; and the year of manufacture.

4.2.1 The containers shall be marked suitably to indicate whether lauryl alcohol used in the production of sodium lauryl sulphate is synthetic or not.

4.2.2 The containers may also be marked with the Standard Mark.

3. REQUIREMENTS

3.1 Description — The material shall be in the form of flakes, powder, needles, granules and crystals or solution. The material in the form of solid shall be white or light yellow in colour and have a faint characteristic odour. The material in the form of solution shall be clear and free from any suspended matter.

3.2 Solubility in Water — A 10 percent solution of the material shall be clear or slightly opalescent at 30°C.

3.3 The material shall also comply with the requirements given in Table 1 when tested accord-

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturer or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 Representative test samples of the material shall be drawn as prescribed in IS : 3958-1984*.

5.2 Tests for determination of sodium lauryl sulphate shall be conducted on each of the individual sample in a set.

5.3 Tests for all other characteristics shall be carried out on the composite sample only.

*Methods of sampling cosmetics (*first revision*).

TABLE 1 REQUIREMENTS FOR SODIUM LAURYL SULPHATE FOR COSMETIC INDUSTRY
(*Clauses 2.1 and 3.3*)

SL NO.	CHARACTERISTIC	REQUIREMENT			METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
		Type 1	Type 2	Type 3	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Sodium lauryl sulphate, percent by mass, <i>Min</i>	94.0	85.0	30.0	A-2
ii)	Unsulphated fatty alcohols, percent by mass, <i>Max</i>	0.5	2.5	1.1	A-3
iii)	Arsenic (as As_2O_3), ppm, <i>Max</i>	2	2	1	A-4
iv)	Heavy metals (as Pb), ppm, <i>Max</i>	20	20	20	A-5
v)	Sodium chloride, percent by mass, <i>Max</i>	1.0	1.5	1.5	A-6
vi)	Sodium sulphate, percent by mass, <i>Max</i>	2.0	6.0	4.0	A-7
vii)	Alkalinity	←→ To pass the test →→			A-8
viii)	pH (1 percent solution)	←→ 7.5 to 9.5 →→			A-9

APPENDIX A

(*Clause 3.3 and Table 1*)

METHODS OF TEST FOR SODIUM LAURYL SULPHATE FOR COSMETIC INDUSTRY

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemical' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF SODIUM LAURYL SULPHATE

A-2.0 Two methods are prescribed, Method A based upon determination of combined sulphur trioxide content shall be the referee method and used in case of a dispute. For routine testing, Method B may be used. Since the molecular mass of the fatty alcohol varies depending on actual composition, the actual molecular mass, as declared by the manufacturer, shall be used in calculation.

*Specification for water for general laboratory use (*second revision*).

A-2.1 Method A

A-2.1.0 *Outline of the Method* — Sodium lauryl sulphate is determined by estimating the combined sulphur trioxide by cationic titration and then calculating the sodium lauryl sulphate content from the value of combined sulphur trioxide obtained.

A-2.1.1 Reagent

A-2.1.1.1 *Cationic reagent* — Weigh 1.5 ± 0.001 g of cetyl trimethyl ammonium bromide (cetab) in a 250-ml beaker. Add 100 ml of water and stir until dissolved. Transfer quantitatively to a 1 litre volumetric flask, mix well and make to volume.

A-2.1.1.2 *Potassium dichromate solution* — 0.01 M; prepared by dissolving 2.942 g of potassium dichromate, dried at 120 to 140°C for about 3 hours, in water and diluting the solution to 1 litre.

A-2.1.1.3 Dilute sulphuric acid — 4 N.

A-2.1.1.4 Potassium iodide solution — 10 percent.

A-2.1.1.5 Sodium thiosulphate solution — 0.1 N (see IS : 2316-1968*).

A-2.1.1.6 Starch indicator solution — Titrate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into 1 litre of boiling water. Boil for 3 minutes. Allow to cool and decant off the supernatant clear liquid.

A-2.1.1.7 Methylene blue indicator solution — Dissolve 0.1 g of methylene blue in 100 ml of water. Transfer 15 ml of this solution to a one litre volumetric flask and add 50 g of sodium sulphate. Add 500 ml of water and 6.8 ml of concentrated sulphuric acid and shake until solution is complete. Dilute to the mark.

A-2.1.1.8 Chloroform**A-2.1.2 Procedure****A-2.1.2.1 Standardization of cationic reagent**

a) Pipette out 50 ml of standard cationic reagent in a beaker and add 25 ml of standard potassium dichromate solution. Heat the beaker to about 90°C to coagulate the precipitate and filter through filter paper (Whatman No. 40 or equivalent). Wash the precipitate thoroughly, collecting the filtrate and washings in a beaker. Add to the beaker, 10 ml of dilute sulphuric acid and 10 ml of potassium iodide solution. Titrate the liberated iodine with standard sodium thiosulphate solution, adding starch solution towards end of titration.

b) Calculate the normality of cationic reagent by the following formula:

$$N = \frac{0.02}{50} \left(25 - \frac{V}{0.6} \right)$$

where

N =normality of the cationic reagent, and

V =volume in ml of standard sodium thiosulphate solution used in the titration in A-2.1.2.1(a).

A-2.1.2.2 Titration of the material

a) Weigh accurately a quantity of the material sufficient to give approximately 0.320 g of combined sulphur trioxide in a 250 ml beaker. (A rough estimate of the quantity required is obtained by dividing 320 by the expected combined sulphur trioxide content.) Use about 700 to 800 ml of warm water to transfer quantitatively to a 1 litre volumetric flask. Warm on a steam bath

and shake gently until the material is dissolved and solution is clear. Cool and dilute to the mark with water and mix thoroughly.

b) Pipette out 10.0 ml of the solution from the volumetric flask into a 100 ml glass cylindrical titration vessel. Add into it 25.0 ± 0.5 ml of methylene blue indicator solution and 10 ± 0.5 ml of chloroform. Titrate with the cationic reagent to the end point, rocking the titration vessel after each addition to avoid formation of emulsion and maintaining the temperature within 20 to 30°C by immersion in water, if necessary. After the first few additions, the blue colour concentrates entirely in the chloroform layer, but as the titration proceeds, there is a slow transfer of colour to the water layer. As the end point is approached, the rate of transfer of colour increases and the cationic reagent shall be added dropwise with vigorous shaking after each addition. If the approximate titration value is known, 80 percent of the required titrating solution should be added before a shaking since this avoids emulsion trouble. Application of vacuum to the titration vessel may help in breaking an emulsion, if formed. The end point is reached when both the layers have same colour intensity. The end point is very sharp and 0.05 ml of the titrant will cause a distinct change in colour distribution at or near the equivalence point.

Note — The titration value should be as near to 10 as possible, values in the range 8 or 12 are acceptable but they shall never be less than 5 or more than 15.

A-2.1.3 Calculation

a) Calculate the combined sulphur trioxide content A from the following formula:

$$A, \text{ percent by mass} = \frac{800.0 TN}{M}$$

where

T = volume in ml of standard cationic reagent used in the titration,

N = normality of standard cationic reagent [see A-2.1.2.1(b)], and

M = mass in g of the material taken for the test in A-2.1.2.2(a).

b) From the combined sulphur trioxide content, calculate sodium lauryl sulphate content as given below:

Sodium lauryl sulphate,

$$\text{percent by mass} = \frac{A(M + 102)}{80}$$

where

A = combined sulphur trioxide content [see A-2.1.3], and

M = actual molecular mass of fatty alcohol as declared by the manufacturer.

*Methods of preparation of standard solution for colorimetric and volumetric analysis.

A-2.2 Method B

A-2.2.0 Outline of the Method — The material is refluxed with dilute hydrochloric acid and the liberated alcohols are extracted with ether and weighed.

A-2.2.1 Reagent

A-2.2.1.1 Dilute hydrochloric acid — approximately 4 N.

A-2.2.1.2 Diethyl ether**A-2.2.1.3 Acetone**

A-2.2.2 Procedure — Transfer about 5 g of the material, accurately weighed, to a long necked 800 ml flask, and 150 ml of water and 50 ml of dilute hydrochloric acid. Heat carefully under a reflux condenser and boil for about 4 hours. Cool, rinse the condenser with solvent ether, collecting the rinsings in the flask. Transfer the contents of the flask to a 500 ml separating funnel. Wash the flask with small quantities of solvent ether adding the washings to the separating funnel. Extract the solution twice successively with 75 ml portions of solvent ether and collect the ether extracts. Evaporate the ether extract on a water-bath almost to dryness, add 5 ml of acetone and evaporate again to dryness. Dry the residue at $105 \pm 2^\circ\text{C}$ for 15 minutes, cool and weigh.

A-2.2.3 Calculation

Sodium lauryl sulphate, percent by mass

$$= \frac{(M + 102)}{M} \left[\frac{M_1 \times 100}{M_s} - M_3 \right]$$

where

M = molecular mass of the fatty alcohols as declared by the manufacturer,

M_1 = mass in g of the residue,

M_s = mass in g of the material taken for the test, and

M_3 = percent unsulphated fatty alcohols in the material as determined in A-3.

A-3. DETERMINATION OF UNSULPHATED FATTY ALCOHOLS

A-3.0 Outline of the Method — From an aqueous solution of the material unsulphated alcohols are extracted out with petroleum ether and then weighed after removal of solvent.

A-3.1 Reagents

A-3.1.1 Potassium Hydroxide Solution — approximately 2 N.

A-3.1.2 Rectified Spirit — 95 percent (v/v), neutral to phenolphthalein.

A-3.1.3 Phenolphthalein Indicator Solution — Dissolve 0.1 g of phenolphthalein in 60 ml of rectified spirit and dilute with water to 100 ml.

A-3.1.4 Petroleum Ether — Boiling range 60 to 80°C .

A-3.1.5 Alcohol Wash Solution — 10 percent aqueous solution of rectified spirit.

A-3.2 Procedure

A-3.2.1 Weigh accurately about 20 g of the material. Dissolve in 50 ml of hot water and transfer to a 250 ml extraction cylinder using sufficient water to bring the volume to about 70 ml. Add potassium hydroxide solution to make the solution neutral to phenolphthalein and then add 3 ml of the solution in excess. Add rectified spirit to bring the volume to about 150 ml. Cool well below the boiling point of petroleum ether.

A-3.2.2 Extract with petroleum ether at least 5 times using 50 ml portions of the solvent. In each extraction, shake vigorously for 30 seconds. Allow to settle well between extractions. Addition of a few grams of sodium dihydrogen phosphate crystals eliminates formation of emulsion. Draw off petroleum ether layers into a small separating funnel and add 30 ml of alcohol wash solution and shake. Draw off and reject the water layer. Repeat washing until aqueous layer is neutral to phenolphthalein. If the washed petroleum ether extract is not clear, filter through a dry filter paper washing it well with petroleum ether.

A-3.2.3 Remove the solvent from the clear petroleum ether extract by evaporation on a steam-bath out of direct contact with steam. A gentle stream of dry, clean, oil-free air or nitrogen shall be directed over the surface of the extract being evaporated. When the volume of the liquid gets reduced to about 25 ml, it shall be transferred to a weighed dish, washing twice with small portions of petroleum ether. The evaporation shall then be continued in the dish. Remove the dish containing the residue from the bath immediately after the last trace of the solvent has evaporated. Dry it in a desiccator and weigh.

A-3.3 Calculation

Unsulphated fatty alcohols,

$$\text{percent by mass} = \frac{100 M_1}{M_s}$$

where

M_1 = mass in g of the residue in the dish, and

M_s = mass in g of the material taken for the test.

A-4. TEST FOR ARSENIC

A-4.0 Outline of the Method — After incinerating the material, the ash is subjected to the Gutzeit test, the stain produced is compared with standard stain.

A-4.1 Procedure

A-4.1.1 Preparation of Solution — Weigh 1 000 g of the material in a platinum dish and incinerate for about two hours at 200 to 250°C. Cool and treat with a mixture of 5 ml of concentrated sulphuric acid and 5 ml of concentrated nitric acid. Take to fumes on a hot plate, cool and make up the volume to 100 ml.

A-4.1.2 Take 10 ml of the solution prepared in A-4.1.1 and carry out the test for arsenic as prescribed in IS : 2088-1983*, using for comparison a stain obtained with 0.002 mg of arsenic trioxide.

A-5. TEST FOR HEAVY METALS

A-5.0 Outline of the Method — The material is ashed and then extracted with a mineral acid. The colour produced by the solution with hydrogen sulphide solution is matched against that produced with standards.

A-5.1 Apparatus

A-5.1.1 Nessler Cylinders — 50 ml capacity (IS : 4161-1967†).

A-5.2 Reagents

A-5.2.1 Concentrated Hydrochloric Acid — conforming to IS : 265-1976‡.

A-5.2.2 Dilute Hydrochloric Acid — 1 : 1 (v/v).

A-5.2.3 Ammonium Chloride

A-5.2.4 Dilute Acetic Acid — 1 N.

A-5.2.5 Standard Lead Solution — Dissolve 1.60 g of lead nitrate in water, add 1 ml of concentrated nitric acid (conforming to IS : 264-1976§) and make up the volume to 1 000 ml. Pipette out 10 ml of the solution and dilute again to 1 000 ml with water. One millilitre of the diluted solution contains 0.01 mg of lead (as Pb).

A-5.2.6 Hydrogen Sulphide Solution — freshly prepared, saturated solution.

A-5.3 Procedure — Heat 1.0 g of the material in a silica crucible in a muffle furnace at 475 to 500°C. Cool and treat the residue with 3 drops of concentrated hydrochloric acid. Evaporate to

dryness over a low flame and heat again in the muffle furnace for 20 to 30 minutes. A clean white ash should result, otherwise the treatment may be repeated. Dissolve the white-ash in 1 ml of dilute hydrochloric acid and wash with several portions of water into an evaporating dish. Evaporate to dryness on a steam bath and dissolve the residue in about 30 ml of water. Transfer the solution to a Nessler cylinder, add 1 g of ammonium chloride and 1 ml of dilute acetic acid. Carry out a control test in another Nessler cylinder using 2 ml of standard lead solution in place of the material and 1 g of ammonium chloride and 1 ml of acetic acid. To each Nessler cylinder, add 10 ml of hydrogen sulphide solution, dilute to the mark and shake well. Compare the intensity of colour produced in the two tubes.

A-5.3.1 The limit prescribed in Table I shall be taken as not having been exceeded if the intensity of the colour obtained with material is not greater than that obtained in the control test.

A-6. DETERMINATION OF SODIUM CHLORIDE

A-6.0 Outline of the Method — Chlorides are estimated by titration against silver nitrate solution.

A-6.1 Reagents

A-6.1.1 Dilute Nitric Acid — 1 : 20 (v/v).

A-6.1.2 Potassium Chromate Indicator Solution — 5 percent solution (see IS : 2263-1979*).

A-6.1.3 Silver Nitrate Solution — 0.1 N (see IS : 2316-1968†).

A-6.2 Procedure — Dissolve about 5 g of the material, accurately weighed, in about 50 ml of water. Neutralize the solution with dilute nitric acid using litmus paper as indicator. Add a few drops of potassium chromate indicator solution and titrate with the silver nitrate solution till a permanent reddish tinge just appears.

A-6.3 Calculation

Sodium chloride,

$$\text{percent by mass} = \frac{5.845 VN}{M}$$

where

V = volume in ml of standard silver nitrate solution used in the titration,

N = normality of standard silver nitrate solution, and

M = mass in g of the material taken for the test.

*Methods of preparation of indicator solutions (first revision).

†Specification for Nessler cylinders.

‡Specification for hydrochloric acid (revised).

§Specification for nitric acid (first revision).

†Methods of preparation of standard solutions for colorimetric and volumetric analysis.

A-7. DETERMINATION OF SODIUM SULPHATE

A-7.0 Outline of the Method — Sodium sulphate is separated from sodium lauryl sulphate using rectified spirit. It is then determined by precipitation as barium sulphate.

A-7.1 Reagents

A-7.1.1 Rectified Spirit — 95 percent (*v/v*), neutral to phenolphthalein.

A-7.1.2 Concentrated Hydrochloric Acid — conforming to IS : 265-1976*.

A-7.2 Procedure — Weigh accurately about 5 g of the material in a beaker. Heat with 200 ml of rectified spirit on a steam bath for 2 hours. The mixture shall be alkaline to phenolphthalein throughout this heating period. Filter the solution and reject the filtrate. Transfer the residue with washing to a beaker, add 200 ml of water and neutralize with concentrated hydrochloric acid adding about 0.5 ml of the acid in excess. If the solution is not clear, it shall be filtered and the paper washed until free from chlorides. The volume of the solution at this stage shall be 250 to 300 ml. Carry out the determination for sulphates as prescribed in IS : 2317-1975†.

A-7.3 Calculation

$$\text{Sodium sulphate (as } \text{Na}_2\text{SO}_4\text{),} \\ \text{percent by mass} = \frac{60.68 M_1}{M_2}$$

*Specification for hydrochloric acid (*revised*).

†Method for gravimetric determination of sulphates.

where

M_1 = mass in g of the precipitate, and

M_2 = mass in g of the material taken for the test.

A-8. TEST FOR ALKALINITY

A-8.1 Reagents

A-8.1.1 Phenol Red Indicator Solution — 0.1 percent solution in rectified spirit (see IS : 323-1959*).

A-8.1.2 Standard Hydrochloric Acid — 0.1 N, accurately standardized.

A-8.2 Procedure — Dissolve 1.0 g of the material in 100 ml of water, add a few drops of phenol red indicator and titrate with standard hydrochloric acid.

A-8.2.1 The material shall be taken to have passed the test if not more than 0.6 ml of standard hydrochloric acid is required for neutralization.

A-9. DETERMINATION OF pH

A-9.1 Apparatus

A-9.1.1 pH Meter — provided with glass and calomel electrodes.

A-9.2 Procedure — Heat 1 g of the sample in 100 ml of water to 80°C. Cool and filter. Determine the pH of the filtrate using the suitable pH meter.

*Specification for rectified spirit (*revised*).

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BUREAU OF INDIAN STANDARDS

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksantha
(Common to all Offices)

Regional Offices :

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg
NEW DELHI 110002 { 331 01 31
{ 331 13 75

Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola
CALCUTTA 700054 37 86 62

Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036 53 38 43

Southern : C. I. T. Campus, IV Cross Road, MADRAS 600113 235 02 16

Western : Manakalaya, E9 MIDC, Marol, Andheri (East)
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